Influence of Preparation Process on Microstructure, Critical Current Density and T_c of MgB_2/Fe/Cu Wires

Y. F. Wu[1]†, G. Yan[1], J. S. Li[2], Y. Feng[1], S. K. Chen[1], H. P. Tang[1], H. L. Xu[3], C. S. Li[1], P. X. Zhang[1], Y. F. Lu[1]

[1] Northwest Institute for Nonferrous Metal Research, P. O. Box 51, Xian, Shaanxi 710016, P. R. China

[2] Northwestern Polytechnical University, Xi'an 710012, P.R.China

[3] School of Material Science and Engineering, Zhengzhou University, Zhengzhou, Henan 450002, Peoples Republic of China

Abstract. The powder-in-tube MgB_2 wires were prepared by high energy milling of Mg and B powder. The powder was not mechanically alloyed for 2h short milling time. However, the MgB_2 grains in wires are very small (20 100nm) and resembled to the dimple after post-heat treatment. The clear evidence for transcrystlline fracture is observed. It indicated that the grain connection was greatly improved and the fluxing pinning was significantly enhanced. Another point to view is no intermediate annealing was adopted during the whole rolling process. The influence of the post-heat treatment on the transport current density was studied. Despite the lower T_c of about 35K, the transport current density reaches to $3\times 10^4 A/cm^2$ at 15K and 3.5T for 700 °c sintered wires.

PACS numbers: 74.70.Ad, 74.62.Bf, 74.25.Qt, 74.25.Sv, 74.50.+r, 74.70.-b

† To whom correspondence should be addressed (wyf7777@tom.com)

1. Introduction

Preparation of MgB_2 wires depends very critically on the precursor powder used for the powder-in-tube (PIT) techniques. Small grain sizes are favored preconditions for achieving high quality wires. Mechanical alloying (MA) technique for MgB_2 powder preparation is expected for obtaining enhanced magnetic flux pinning by microstructure refinement. However, it costs as long as 20 100h[1,2,3-8] for in situ MA precursor powder preparation. The use of short-time unalloyed high energy milling of Mg and B powder as precursor material represents an efficient combination between conventional powder preparation and mechanical alloying techniques. Fe sheath was considered as one of suitable materials however hard it is. It is also gradually recognized that repeated annealing during rolling process would cause the inevitable diffusion of oxygen into the filament of MgB_2 wire and decrease J_c greatly. Therefore, how to reduce intermediate annealing steps during material preparation become one of the biggest challenges as literatures[8-10] referred to.

2. Experimental details

Mg(99.8%) and amorphous B (95%) powder with 5% Mg surplus were filled under purified Ar-atmosphere into an agate milling container and milling media. The milling was performed on a SPEX 8000M mill for 2h using a ball-to-powder mass ratio of 3. Monofilamentary wires were prepared by conventional PIT method. The powder was packed into coaxial Cu/Fe tubes (outer diameter 14 mm and inner diameter 7 mm) forming the billets. Next, billets were groove-rolled and drawn to a wire diameter of 1.2mm without any intermediate annealing. The post heat treatment was performed at different temperatures for 1 hour under ultra-high purity Ar-atmosphere. The phase content of MgB_2 was analysed by x-ray diffraction scans performed on a Philips APD1700 diffractometer with Cu K radiation. The surface morphology and microstructures of the samples were characterized by JSM-6700F scanning electron microscope. The superconducting transition temperature, T_c, was obtained by resistance-temperature method. The critical currents were evaluated from V-I curves taking a 1V cm^{-1} criterion.

3. Results and discussion

As we know, the ductile deformation behavior at low temperatures is favored for acquiring high J_c performance MgB_2 wires. In our study, no intermediate annealing was adopted during the whole rolling process. The total working modulus of Fe sheath exceeds 99 percent. A typical cross section for wire diameter of 1.2 mm is shown in figure 1. We concluded from our experience that the reduction in pass and rolling rate during rolling process should be controlled to a lower lever.

The microstructures of samples sintered at different temperatures are shown in

Fig.2. The Scanning electron microscope images mainly show dimple-like grains for 700 800 °c sintered samples. The grain sizes are about 20 100nm for 700 °c and 750 °c sintered samples. The clear evidence for the transcrystlline fracture is observed. It indicated that the grain connectivity was enhanced greatly. The grains are grown up to 100 250nm for 800 °c sintered sample. While for 650 °c reacted sample, large plate-like column crystals were observed. The impurity phases are evidently observed for sample sintered at 800 °c. Fig.3 shows the wrap-around distribution of the second phases of the 800 °c sintered sample. EDX analysis indicates that the bright one is O-rich zone and the grey one is Mg-rich zone,as seen in fig.4.

Fig.5 indicates the superconducting transition T_c by resistance measurement for the samples sintered at different temperatures. As we can see, all samples have sharp transitions. T_c of the high energy milling samples ranged from 34.5 to 35.5 K, which is much lower than 39K. Most likely, this is not due to a deviation from the ideal stoichiometry of the superconducting compound[2]. From x-ray analysis, interaction between the constituents of precursor powder and the sheath material can also be excluded. It seems that the suppression of T_c is caused by oxygen contamination in grain boundary induced by high energy milling as well as sintering process.

Fig.6 shows the J_c-B curve of the MgB_2 wires at different temperatures. The critical currents were evaluated from V-I curves taking a 1V cm^{-1} criterion. As we can see, the critical current densities are influenced greatly by heat treatment. The higher or lower heat treatment induced second phases and inferior microstructure, which led to bad grain connectivity and contribute to dramatic decrease of J_c. In the lower field, the MgB_2 wires sintered at 700 °c show the higher critical current density. In the higher field, the MgB_2 wires sintered at 750 °c show the higher critical current density. The critical current density reaches to $3\times 10^4 A/cm^2$ at 15K and 3.5T for the wire sintered at 700 °c.

In Summary, we succeeded in preparing high critical current density MgB_2 wire using unalloyed high energy milling precursor powder. The powder preparation process was greatly shortened. It demonstrated that it is an effective approach to get fine crystalline MgB_2 with good grain connectivity and high J_c performance. The critical current density reaches to $3\times 10^4 A/cm^2$ at 15K and 3.5T for the wire sintered at 700 °c.

4. Acknowledge

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Figure captions

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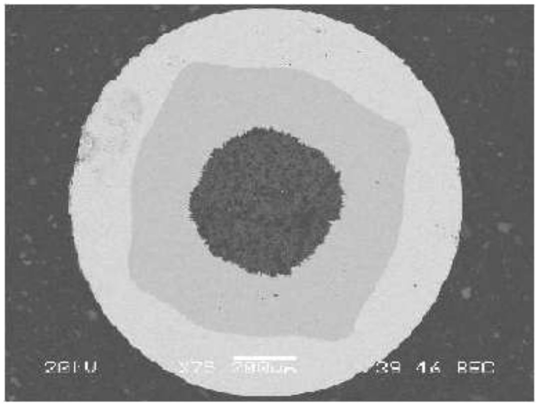

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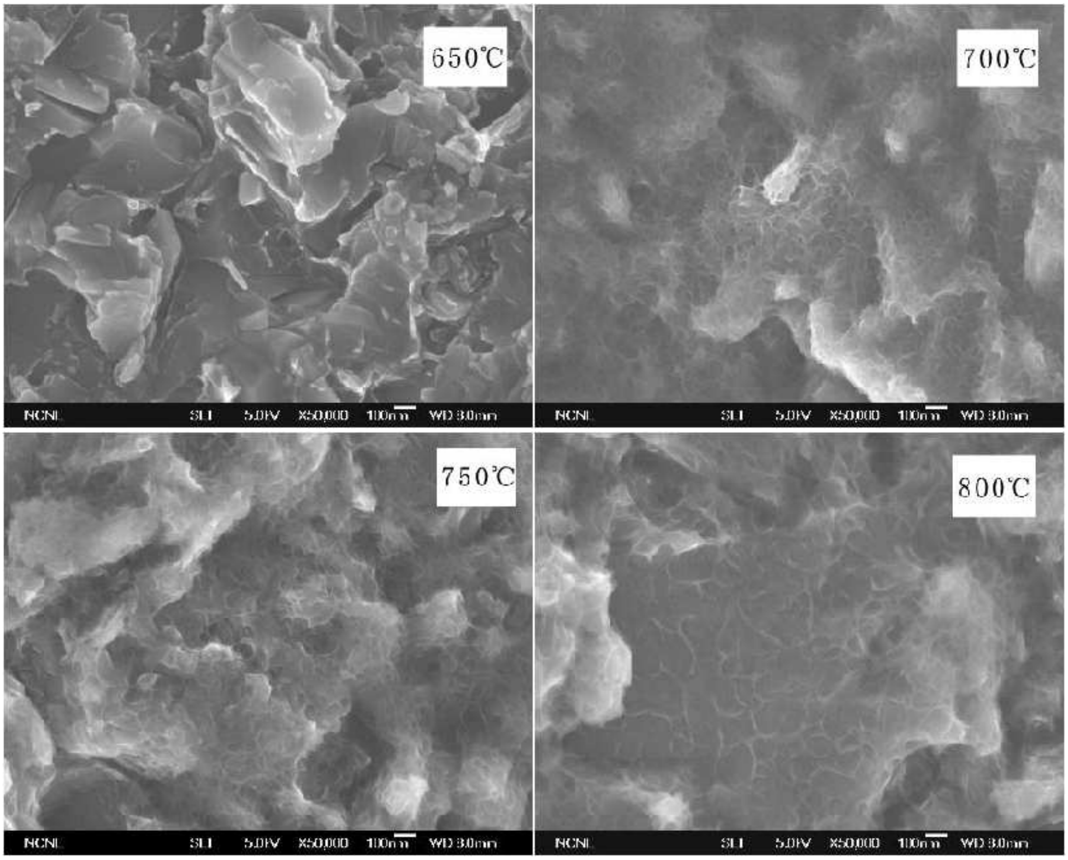

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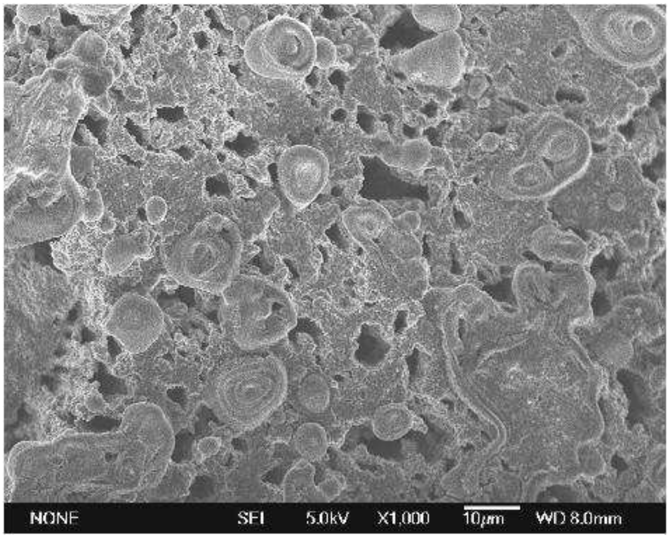
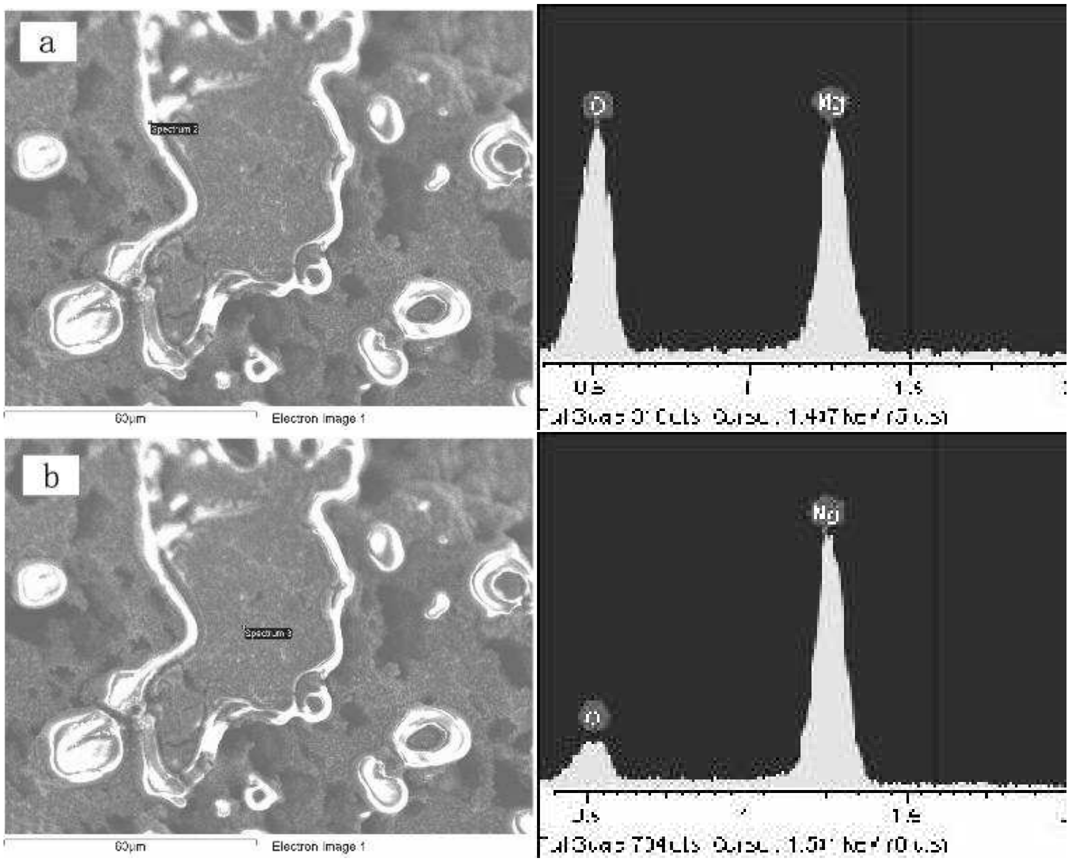


Fig.4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60μm
Electron Image 1
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Mg
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1
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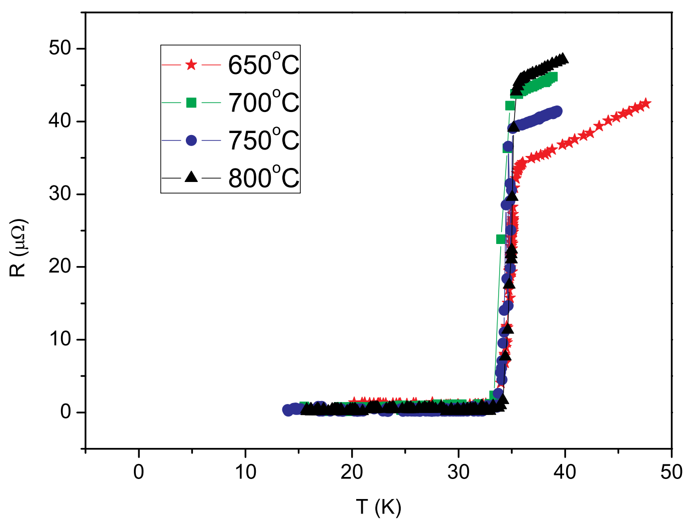
b
Spectrum 3
60μm
Electron Image 1
O
Mg
0.5
1.5

Fig.5



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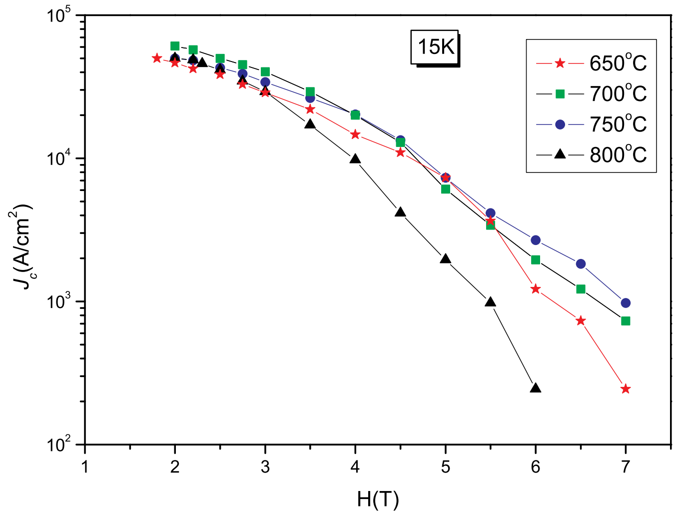

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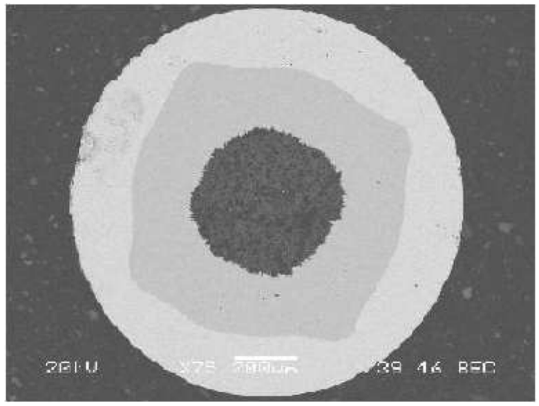

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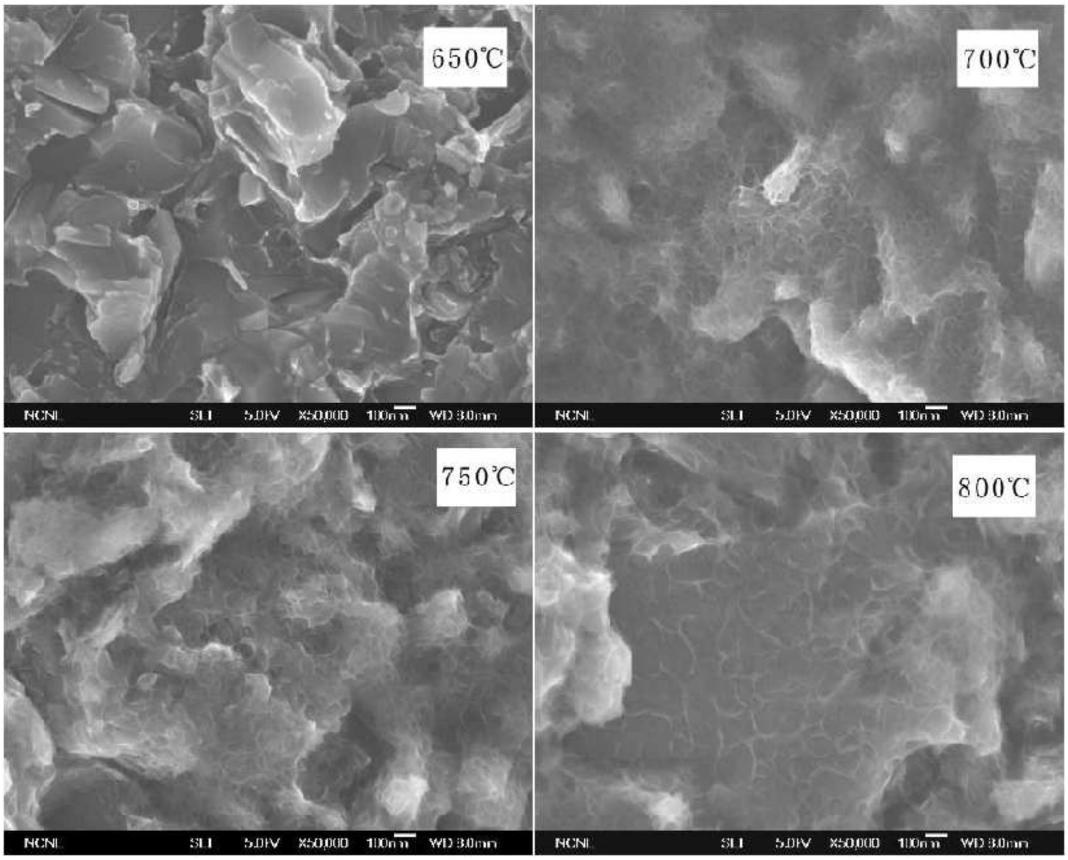

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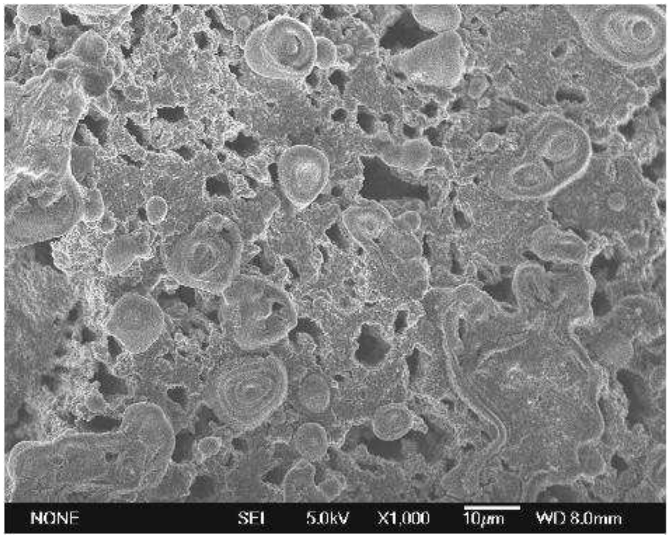

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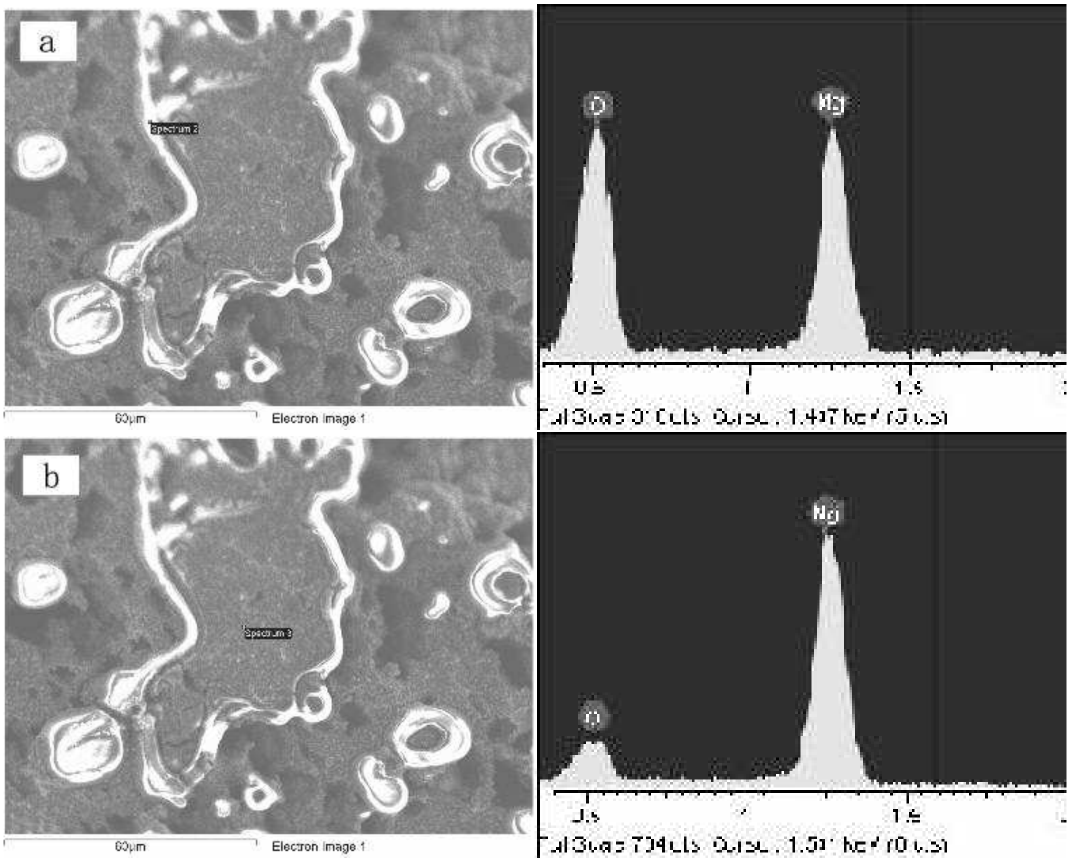
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Electron Image 1
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Mg
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b
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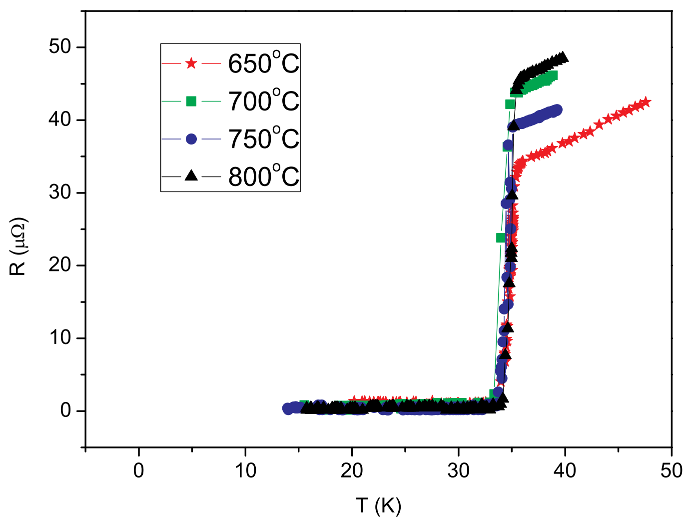

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